Solver EC

Solver EC

Solver EC Application

Purpose:

In-situ control/modification of the surface morphology of single crystal and polycrystal electrodes (samples) during electrochemical process (in situ) in electrolyte solutions together with STM (max resolution – atomic).

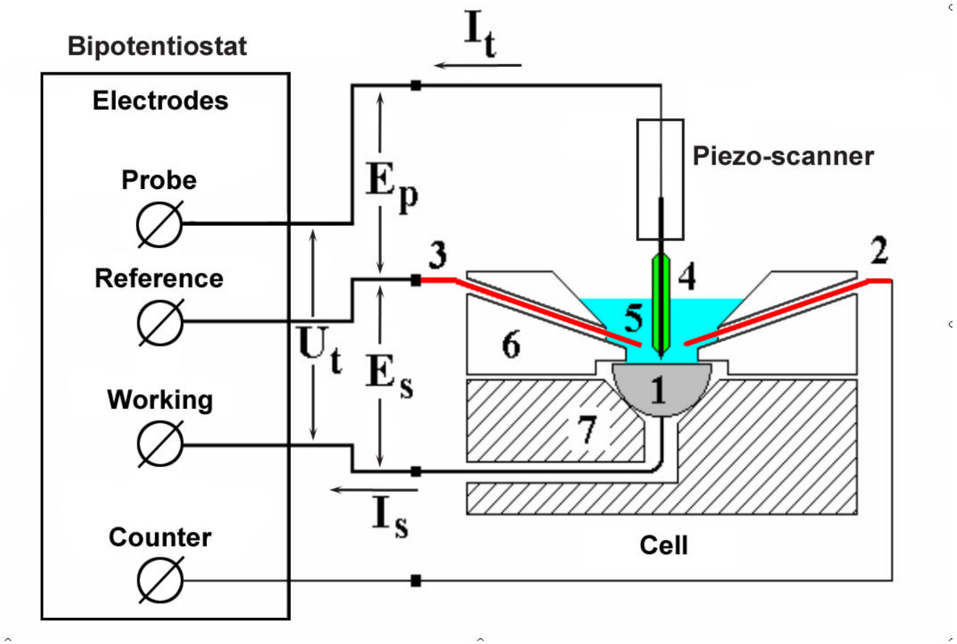
Solver EC Application

Best choice to study:

forming of electrochemical adsorption layers process,
metal electrodeposition and electrodissolution,
electrocorrosion and electroreduction process, oxidations process for different metals or other conductive materials,
electrically nanoscale active centres, ions and molecules
mass transfer process, electrode/electrolyte interface,
electrochemical lithography operations and etc.

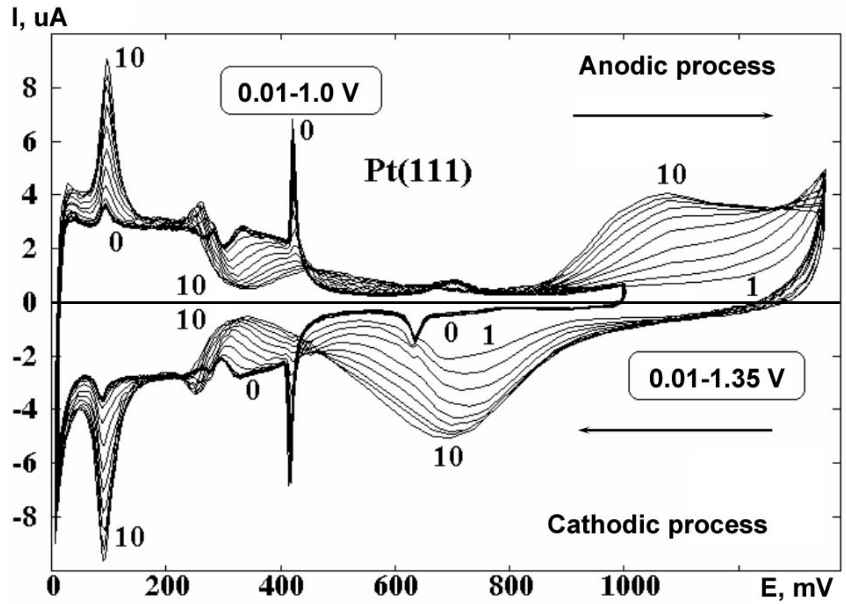
Solver EC Basic Principles

Basic Working Scheme



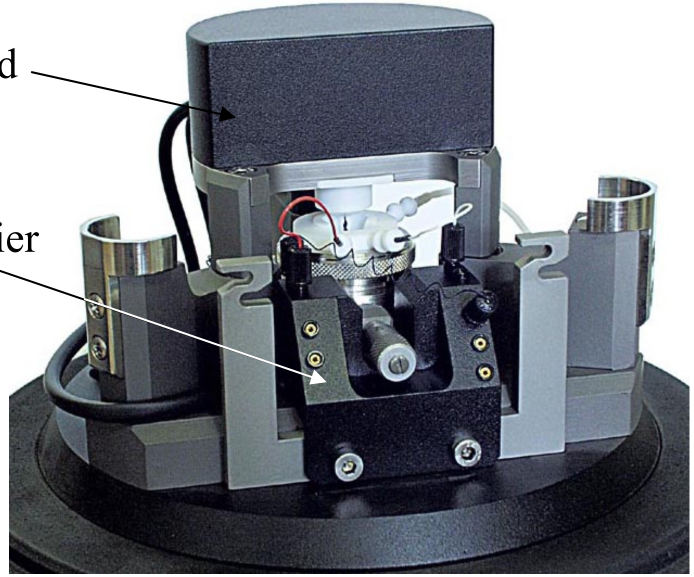
1. Sample
2. Counter Electrode
3. Reference Electrode
4. STM Probe
5. Electrolytic Solution
6. Cell
7. Cell Base

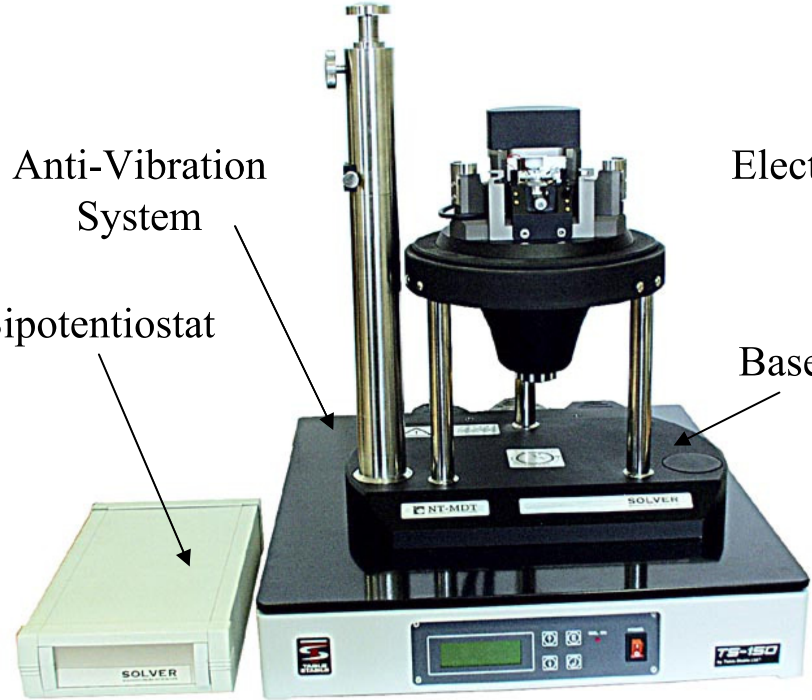
Cyclic voltammetry of Pt (111) in 0.05 M H_2SO_4 + 1 mM $CuSO_4$ solution

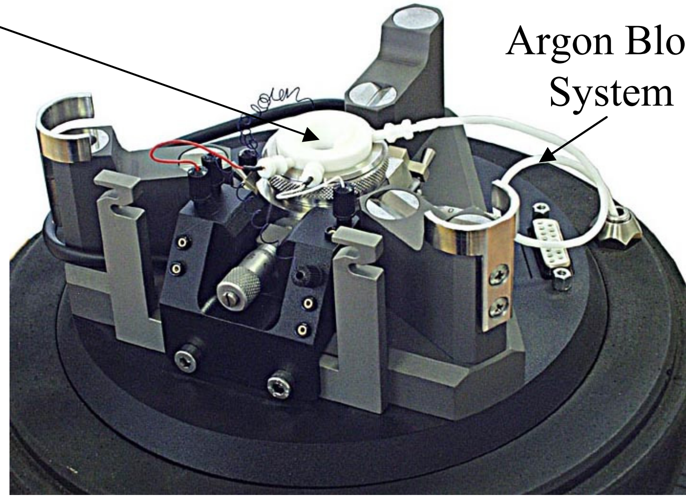


Solver EC Basic Configurations

- Scanner Head
- Bipotentiostat
- Preamplifier
- Electrochemical Cell
- Argon Blow System
- Base (Solver Pro, Solver P47H-Pro)
- Controller
- Anti-Vibration System



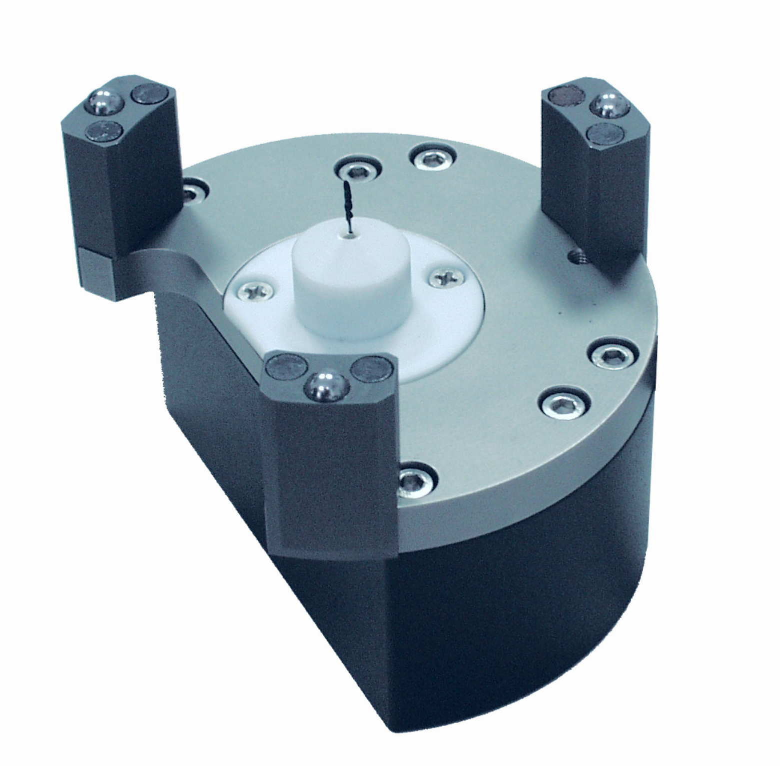




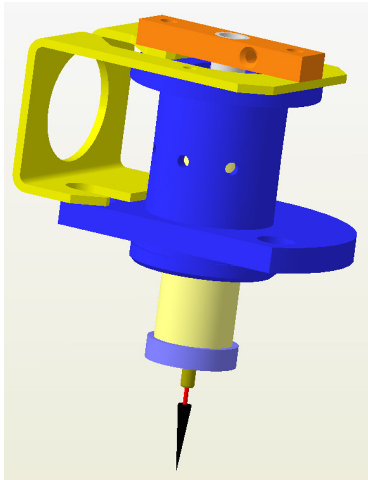
NT-MDT
Molecular Devices and Tools for NanoTechnology

Your choice is granted

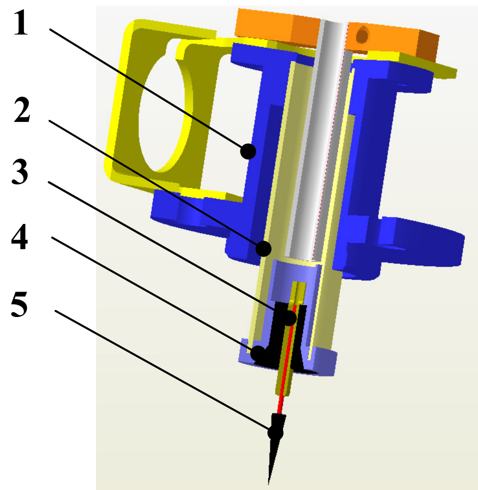
Solver EC. Scanner Head & Scanner Block



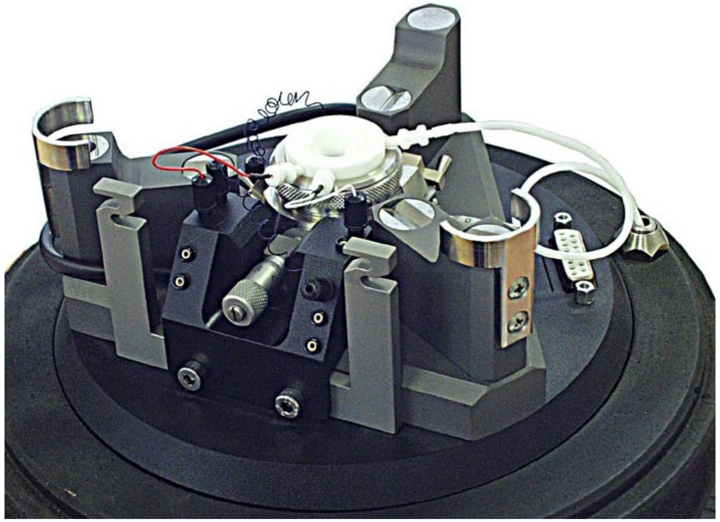
- Scanner : 7×7×1.5 µm (optional - 18×18×2.5 µm), (future – 3x3um, 50x50um)
- Scanning by Tip
- STM Tips Holder
- Preamplifier: ± 50 nA, noise 3 pA



1. Base
2. Piezotube
3. Tip Holder
4. Insulator (ceramic)
5. Insulated Tip

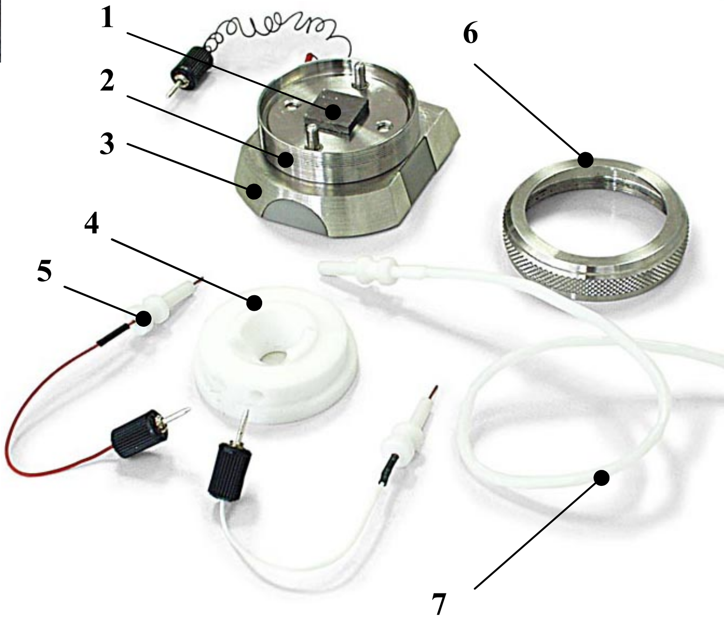


Solver EC. Electrochemical Cell



- Cell material: Teflon
- Platform: Standard orthogonal
- Max size: ∅35×10 mm
- Electrolyte volume: 0.5 to 1.5 ml
- Argon blow system

1. Working Electrode (sample)
2. Cell Base
3. Platform
4. Cell
5. Electrode Holder
6. Fixing Ring
7. Teflon Tube for Gas (Ar) Input



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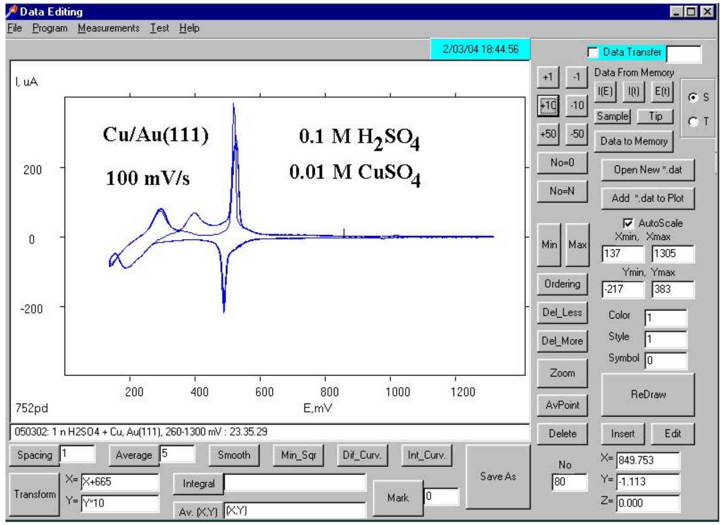
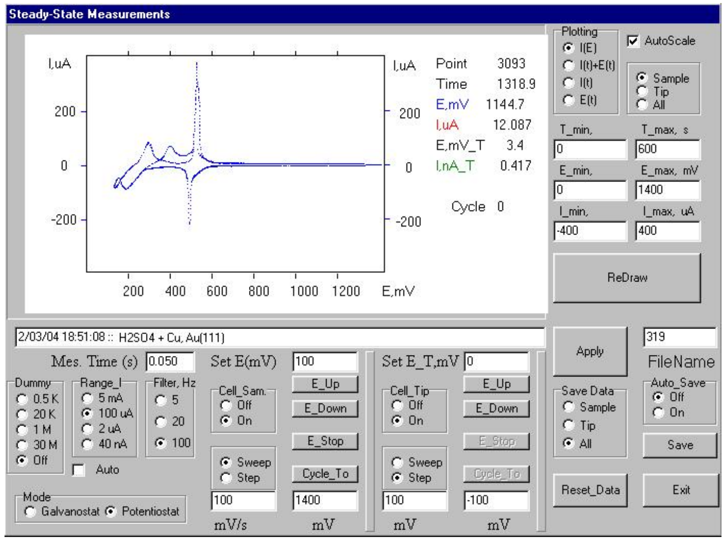
Solver EC. Bipotentiostat & Software





Bipotentiostat

- Output Compliance Voltage: ± 15 V
- Applied Voltage Range: ± 5 V (stability ± 1 mV)
- Current Ranges: ± 5mA; ± 100 µA; ± 2 µA (accuracy of measurement ±0.1 % of the range)
- Reference Input Impedance: > 10^{11} Ω

Software

- Modes: *manual*; *sweep* (1 mV/s to 100 mV/s), *pulse* (time resolution 10 ms); *potentiostatic current transients*; *cyclic and linear voltammetry*; *universal programmer*
- Computer: Pentium II or higher
- System: Windows 98/XP

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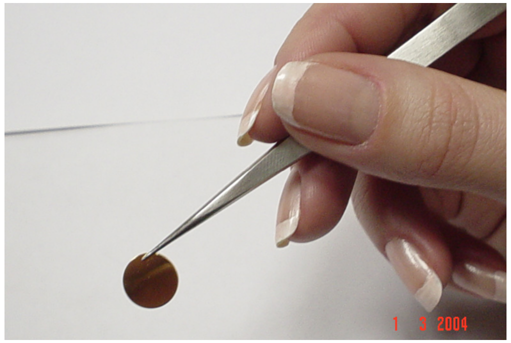
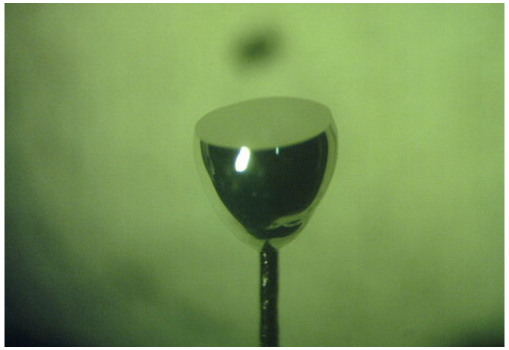
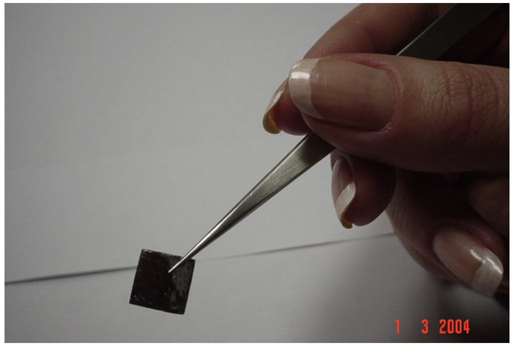
Solver EC. Electrodes



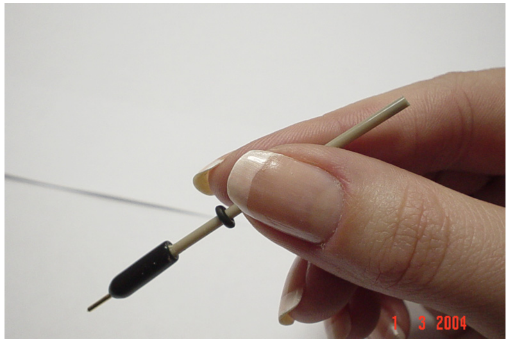
Working Electrode:
monocrystal Au (111)



Working Electrode:
monocrystal Pt (111)



Working Electrode:
HOPG (0001)



Reference Electrode:
Ag/AgCl (Cypress System)

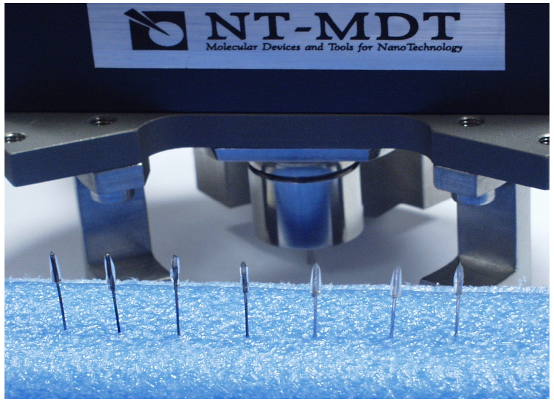
Working:
∅ 4-15 mm (Au 111),
Pt (111), HOPG, polished
polycrystalline metals)

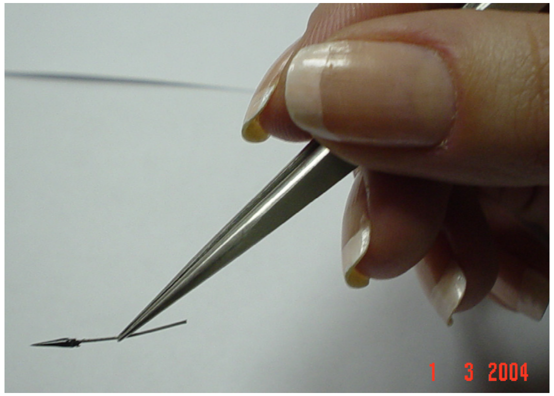
Counter: Cu, Pt, Au-wire

Reference: Cu, Pt, Au-wire,
Ag/AgCl (Cypress system)

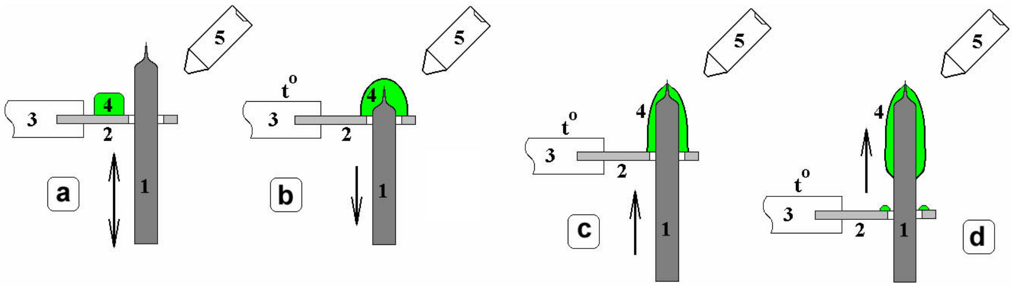
Electrolyte:
H_2SO_4 (0.01 M) + $CuSO_4$ (0.01M)
or any suitable electrolyte

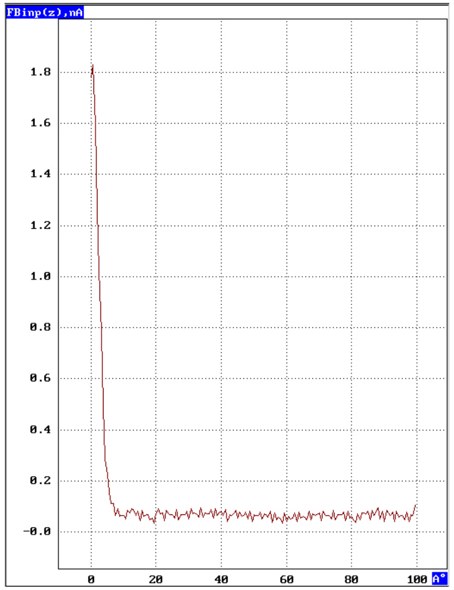
Solver EC. STM Electrochemical Tips





Technological Scheme of Tip Insulation Process



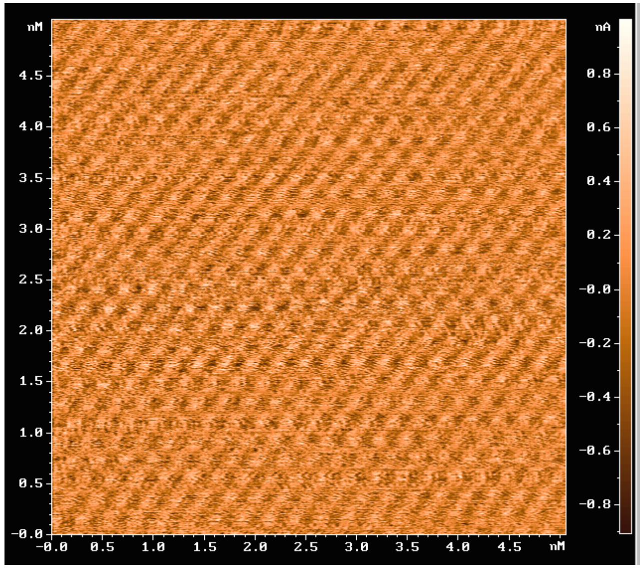


Probe Spectroscopy

- Material: W, Pt-Ir (10-25%)
- Size: ∅ 0.4-0.5 mm,
  max length 20 mm
  (insulated part 5-7 mm)
- Sharpening: electrochemical, mechanical
- Insulation: “Apiezon Wax”, polyethylene

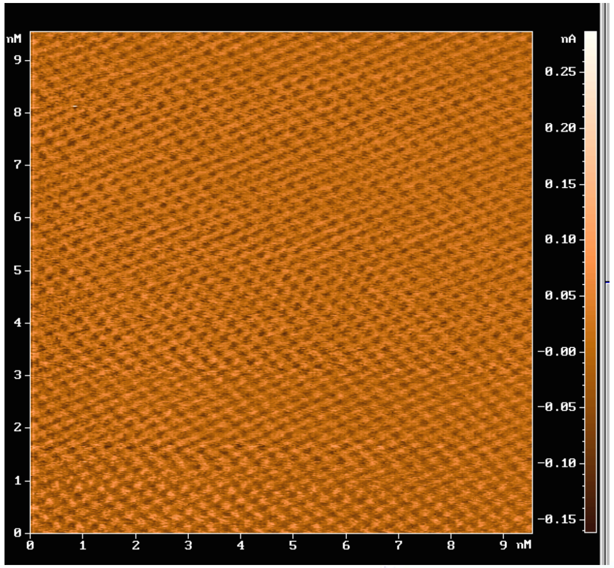
Solver EC. Results

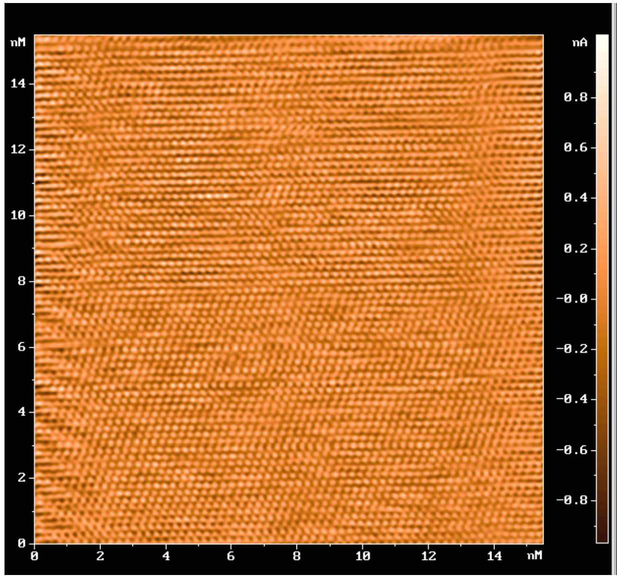
STM Image of Graphite (on air)



Scan size: *5 × 5 nm*

Scan size: *10 × 10 nm*

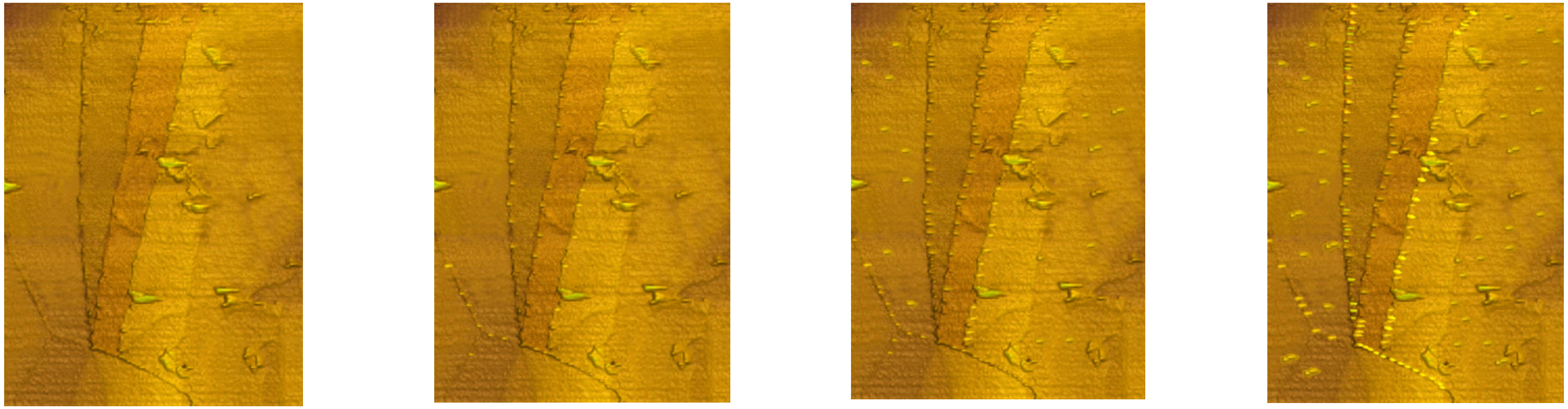




Scan size: *15 × 15 nm*

Solver EC. Results

STM Image of Graphite (in solution)



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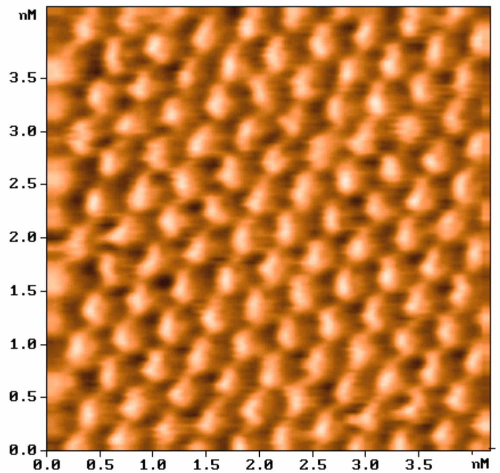
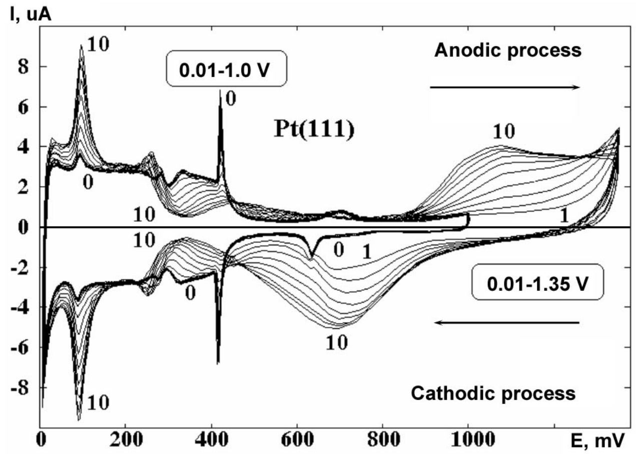
Scan size: *0.8 × 1.1 µm*
Graphite surface decoration by copper clusters during dynamic electro deposition process

Scan size: *4.1x4.1 nm*
Atomic resolution of HOPG in Electrolyte solution (0.05 M H2SO4 + 1 mM CuSO4) under potential.



Solver EC. Results (Cu/Pt)



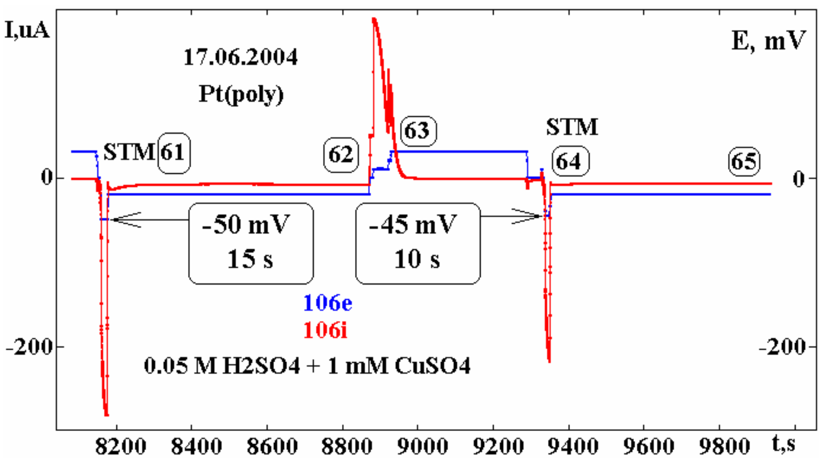
Cyclic voltammogram of the Pt electrode

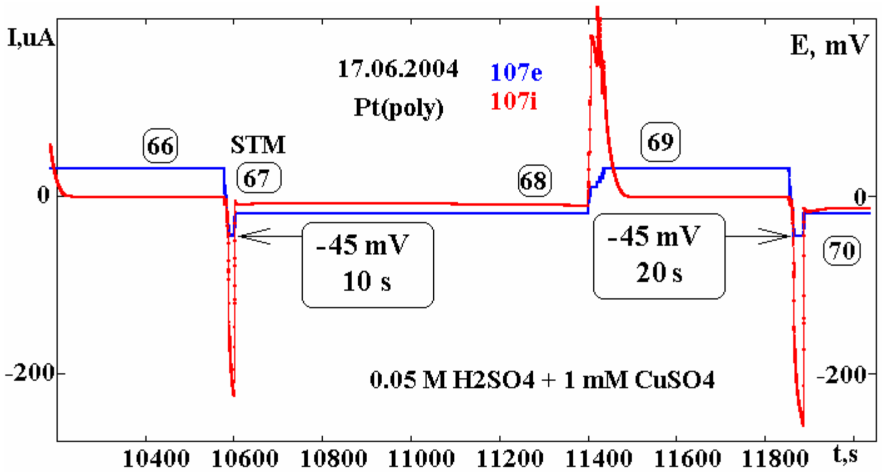
A piece of Cu wire was used as the quasi reference electrode and counter electrode. Tungsten tips, which were etched in 2M KOH and subsequently coated with “Apiezon Wax” were used as tunneling probes. The Faradaic background current through the tip was below 10 nA.The Pt poly crystal was 3 mm in diameter. Prior to each experiment, the Pt electrode was flame annealed in flame for a few minutes.

The electrolyte was 50 mM H_2SO_4 containing 1 mM $CuSO_4$. Periodically the electrolyte was changed at the upper and lower potential limits. After this procedure the voltammetric response of the electrode was in reasonable agreement with the commonly accepted voltammogram of the Pt in 50 mM H_2SO_4.

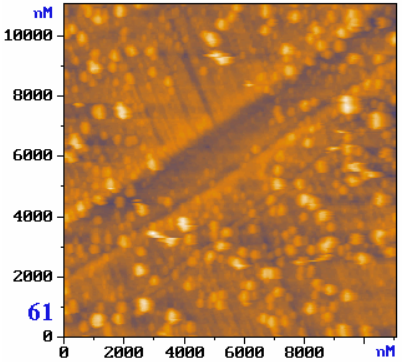
Applied potentials and numbers of STM images are presented in figures below.

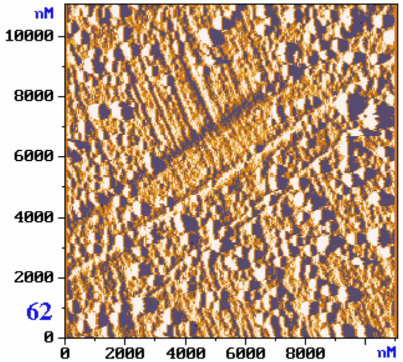
STM images are shown on next slide.

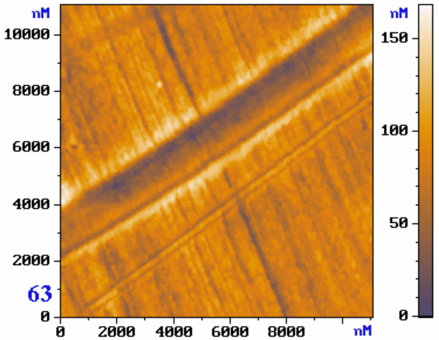


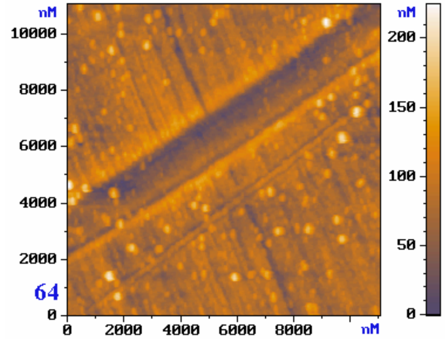


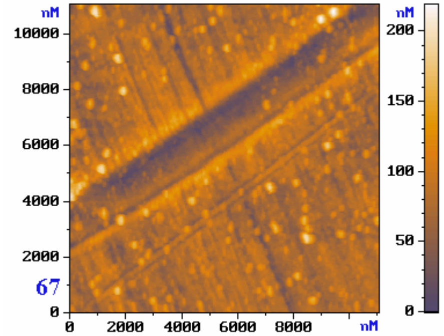
Solver EC. Results (Cu/Pt)

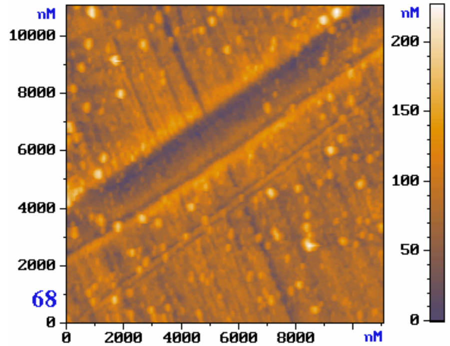


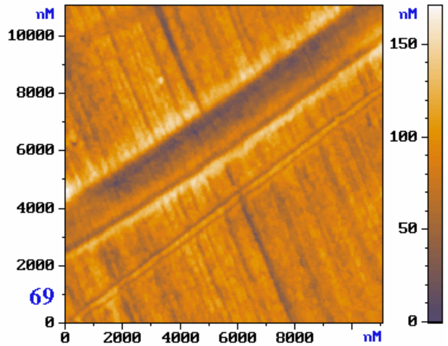








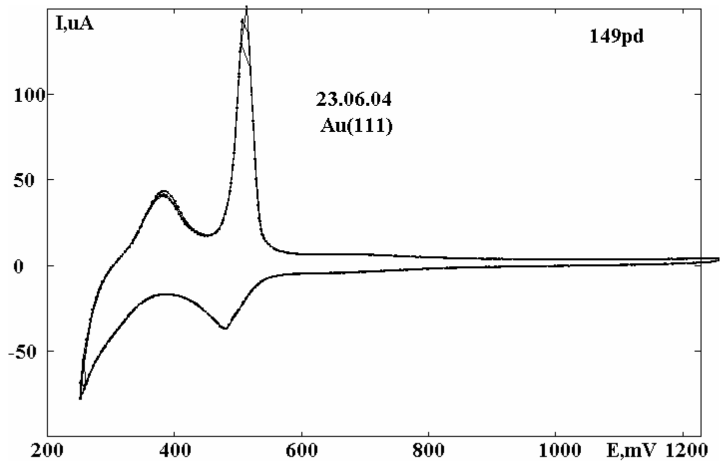




STM images (61) and (62) show nucleus growth. These images were recorded at -50 mV.
STM image 63 shows nucleus dissolving at +30 mV ("clear surface").

STM images (64), (67) and (68) show nucleus growth at other potential (-45 mV). It can be seen that number of nucleus are considerably reduced. Nucleus arrangement are changed.
STM image (69) shows nucleus dissolving at +30 mV ("clear surface").

Solver EC. Results (Cu/Au)

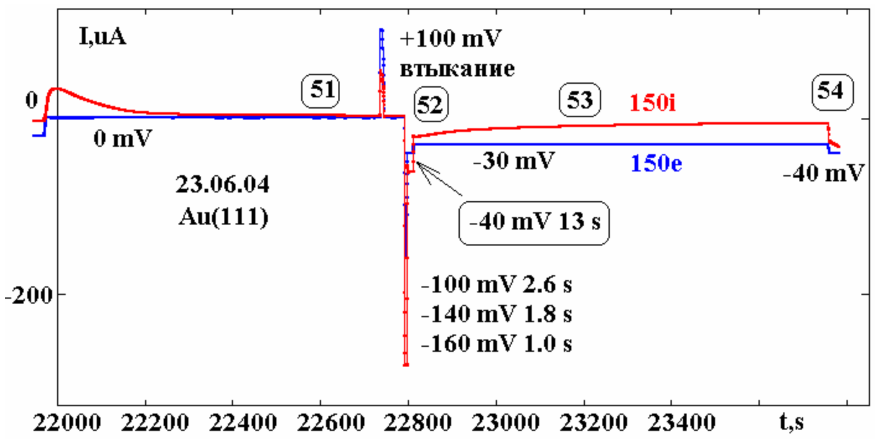


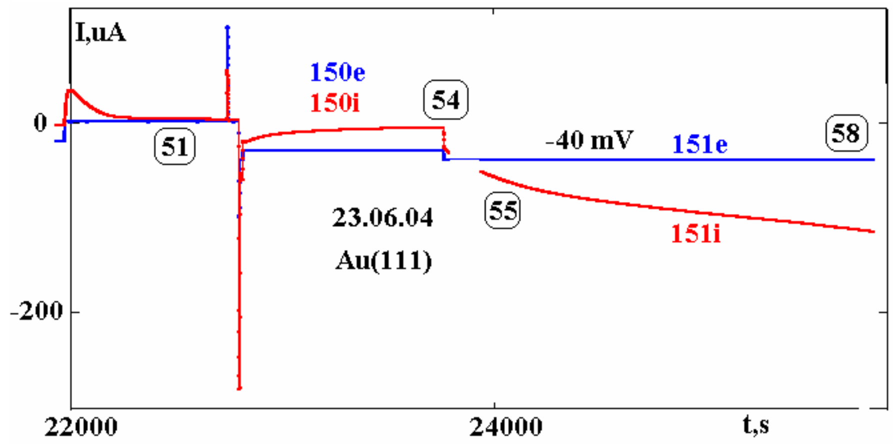
Cyclic voltammogram of the Au electrode

A piece of Cu wire was used as the quasi reference electrode and counter electrode. Tungsten tips, which were etched in 2M KOH and subsequently coated with “Apiezon Wax” were used as tunneling probes. The Faradaic background current through the tip was below 10 nA. The Au monocrystal was 10 mm in diameter. Prior to each experiment, the Au electrode was flame annealed in flame for a few minutes.

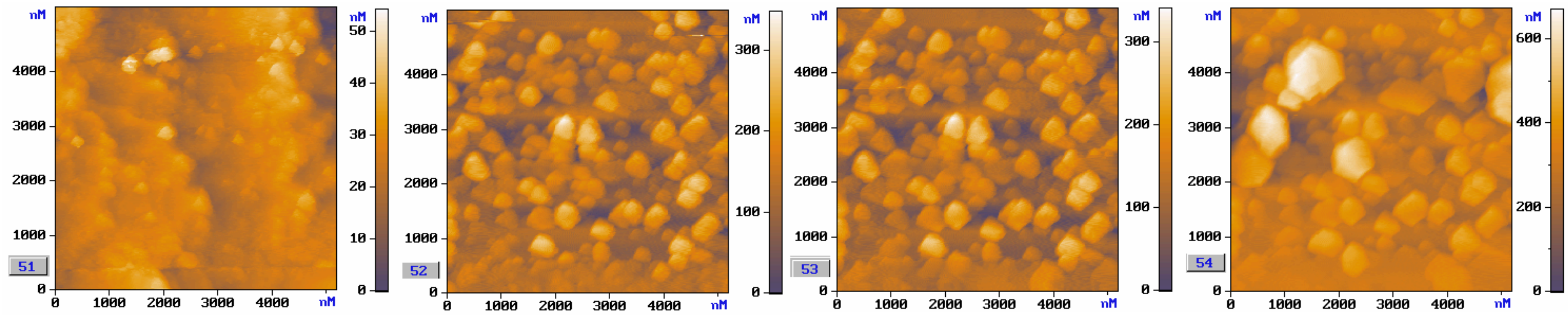
The electrolyte was 50 mM H_2SO_4 containing 1 mM $CuSO_4$. Periodically the electrolyte was changed at the upper and lower potential limits. After this procedure the voltammetric response of the electrode was in reasonable agreement with the commonly accepted voltammogram of the Au in 50 mM H_2SO_4.

Applied potentials and numbers of STM images are presented in figures below. STM images are shown on next slides.

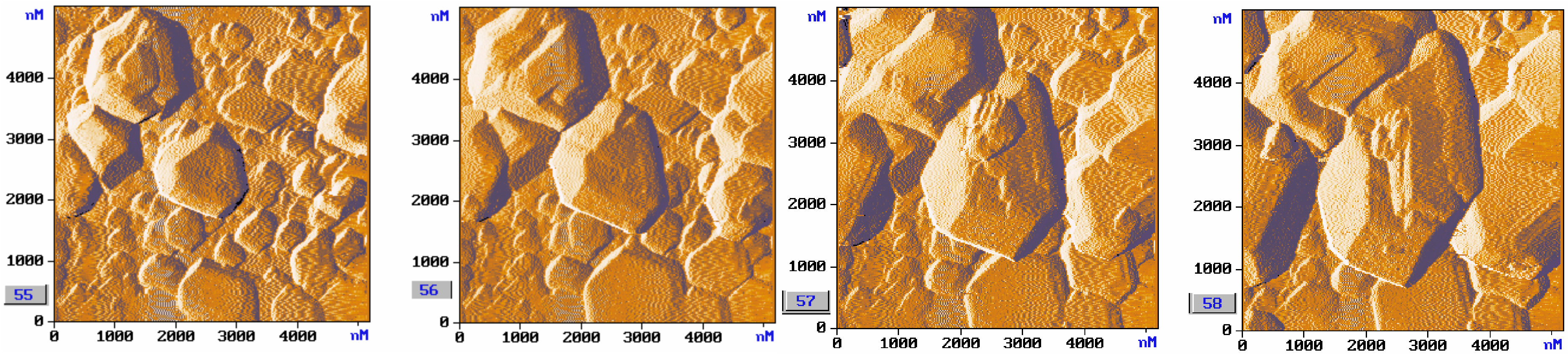




Solver EC. Results (Cu/Au)

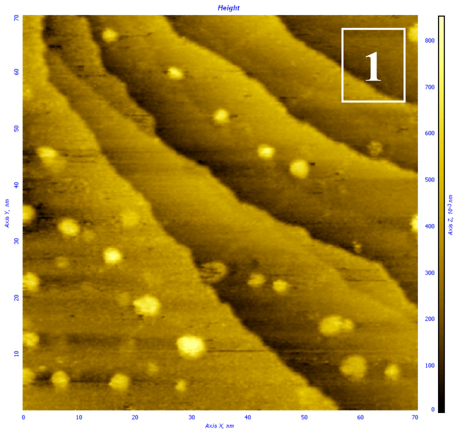


STM Images (51)-(54) show Cu dissolving. There are fair amount small nucleus. Their size is 0.2-0.4 µm.

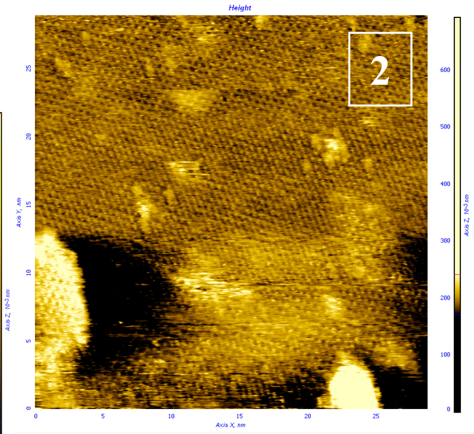


STM Images (56)-(58) show Cu monocrystals growth. It can be seen facets of Cu (111) monocrystal. There are growth steps on big crystals.

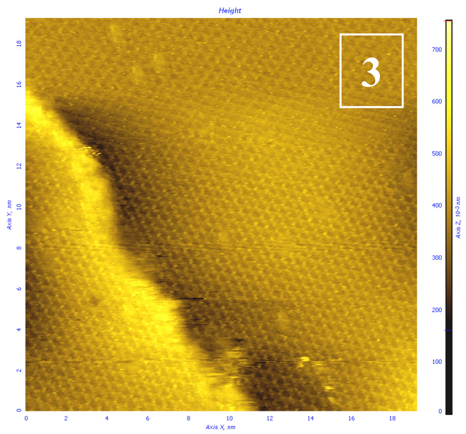
Solver EC. Results (Cu/Au)



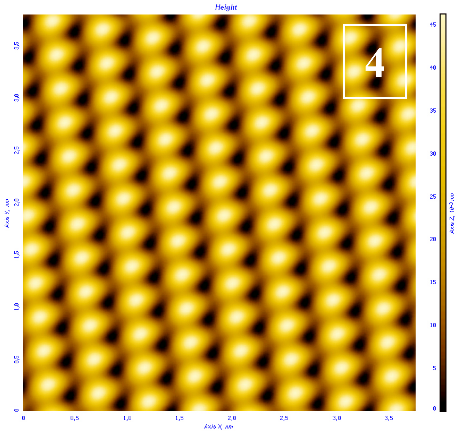
Scan size 70 × 70 nm



Scan size 30 × 30 nm



Scan size 20 × 20 nm



Scan size 3.5 × 3.5 nm

3D

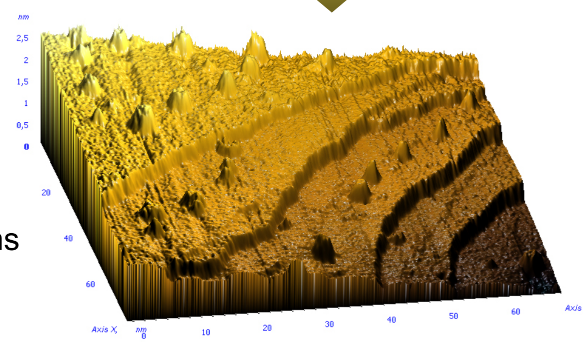


Fig. 1. Au monatomic steps and islands resulting after flame annealing of Au substrate.
Fig. 2, 3, 4. Lateral atomic resolution of co-adsorption lattice (√3x√3)R30° of Cu-ad atoms and bisulphate anions.

Solver EC Highlights

1. Easy set up and clearance TEFLON cell.
2. Flexible design allowing various modifications.
3. Cell design allows mounting a wide range of samples with different thickness and shapes (hemispherical is possible).
4. Insulating medium (purified inert gas) is used to prevent electrolyte contamination.
5. Sample is separated from the piezotube to prevent any scanner damage.
6. A wide range of electrode types.
7. EC device is based on the Solver SPM base and NTegra.
8. High resolution (up to atomic)

THANK YOU!

RMI
ANALYTICAL & TESTING INSTRUMENTS

NT-MDT
Molecular Devices and Tools for NanoTechnology

YOUR CHOICE IS GRANTED!

E-mail: sale@rmi.cz